

RESEARCH ON THE LOW-TEMPERATURE DECONTAMINATION OF SOIL

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INTRODUCTION

Decontamination of organically contaminated soil requires at least two processes: 1) separation of the contaminants from the soil and 2) destruction or stabilization and recovery of the organic contaminants. Techniques used to convert the organic contaminants must produce environmentally acceptable products. A process is seen as more favorable if the inorganic portion remaining after destruction of the organic contaminants retains a "natural" state, since ideally the residual should be returned to the original ecological system.

Low-temperature plasma (LTP) processing has the potential to offer decontamination equivalent to incineration with decreased production of harmful by-products and without changing the morphology of the inorganic matrix. Low-temperature plasmas are formed by electric discharge (rather than thermal means) at conditions that do not result in thermal equilibrium. It is possible with an LTP system for typical plasma reactions to occur at nearly ambient bulk temperatures.

The purpose of this project is to perform the appropriate engineering evaluations needed to scale up an LTP soil decontamination process developed at the Energy & Environmental Research Center (EERC). The LTP process under development uses oxygen plasma, resulting in oxidation of the contaminant species. Previous research at the EERC proved that the technique successfully separates organic contaminants from soil. The success of this previous research indicated that the use of LTP as a decontamination technique should be investigated further, with possible scaleup to real-world application.

METHODOLOGY

A program plan consisting of four phases was developed to enable the design and construction of a real-world-scale prototype unit. The Phase 1 work included operation of the existing EERC LTP unit with integrated gas analysis to verify that the fate of all organic species placed in the reactor could be determined. Phase 2 consists of gathering the engineering information needed for prototype design, while Phase 3 consists of the design, fabrication, and shakedown testing of the prototype. The design, fabrication, and integrated shakedown testing of any add-on equipment necessary for decontamination using the prototype constitute the Phase 4 work effort. This paper summarizes the testing performed during the Phase 1 carbon fate study.

The Phase 1 testing was performed on the EERC bench-scale LTP system. The system consists of a radio frequency (rf) generator, a reactor center, a vacuum system, a feed gas system, and an elaborate product gas collection system. The rf generator and the reactor center were manufactured by the International Plasma Corporation. The quartz reaction chamber is 8 inches long and 4 inches in diameter and has been modified to contain the hardware necessary for agitation during operation. A pair of rotating aluminum trays was used to agitate the sample during operation. The trays each have a lip on one longitudinal edge and are aligned so that, when turning on the rotary coupling, the sample falls from one tray to the other through the plasma. A vacuum pulls the plasma-producing gas through the chamber, while the rf energy is transmitted across the electrodes surrounding the sides of the reaction chamber.

The product gas collection system was designed and fabricated for use in all phases of the project. The system consists of a bypass loop, two product gas collection bags or bladders contained in two vessels, and three syringes that are used to transfer the gas from the collection bags to the analytical system. An on-line gas chromatograph (GC) was available for analysis of the product gas stream during the Phase 1 testing.

A statistically designed experimental matrix was used to gather data concerning the fate of the carbon species. The matrix was designed to vary five independent variables: oxygen flow rate, reactor pressure, rf power, reaction time, and percent carbon in sample. The statistical analysis of the data will indicate which operating parameters affect contaminant removal and destruction efficiencies and how these efficiencies are affected. The matrix was designed so that the test points would be performed in random order to minimize skewing of data that can occur when one variable is held constant for several tests in a row. Tests were performed in blocks to determine whether significant changes occurred in the system over the course of the testing.

All tests were performed using decolorizing carbon as the contaminant source and washed, 60- to 100-mesh sand as the inert matrix. Pure oxygen was used as the plasma-producing gas. The use of carbon and pure oxygen limited the various products of incomplete combustion (PICs) that could be produced, thereby greatly simplifying the product gas analysis. Both the feed and product solid samples were analyzed for carbon, hydrogen, nitrogen, sulfur, and ash content. Feed and product gas samples were analyzed using gas chromatography.

RESULTS

The purpose of this study was not to maximize contaminant removal and destruction efficiencies, but rather to lay the foundation for the testing that will take place during the remaining phases. The goals of the Phase 1 testing included verification that:

- Mass balance closures of $\pm 5\%$ are possible.
- The analytical scheme that is in place is capable of accurate results.
- The fate of the contaminant species can be accurately tracked.
- PICs can be identified and quantified with the existing product gas collection system and the on-line GC.

A mass balance closure criterion of $\pm 5\%$ was chosen as a project quality assurance objective because data gathered from tests with worse mass balances would not accurately represent the tests. Therefore, as a first step, a mass balance was calculated for each test, and those tests not meeting the criterion were repeated. A total of seven tests were repeated based upon initial mass balance calculations. A change in the mass balance calculation was made to account for minor contamination by air, resulting in only two tests that did not meet the mass balance closure criterion. The results of the Phase 1 testing are summarized in Table 1. Table 1 clearly shows that mass balances meeting the criteria were easily obtained. The average mass balance was 100.46%.

Quality assurance (QA) check samples were randomly submitted to verify that the analytical results accurately described the samples. Analyses of both the solid and gaseous products agreed with the calculated values well within the error range of the analytical instrumentation. Average differences between the calculated values and the analyzed values for the solid samples were +0.05 wt% carbon, -0.004 wt% hydrogen, -0.002 wt% nitrogen, +0.084 wt% sulfur, and -0.20 wt% ash. Instrument error is ± 1.0 wt% for ash and ± 0.5 wt% for the other components. The gas analysis checks were equally accurate, with average

TABLE 1. SUMMARY OF DATA GATHERED DURING PHASE 1 CARBON FATE STUDY*

Test No.	Reaction Time (min)	Reactor Pressure (torr)	RF Power (W)	O ₂ Flow Rate (gmol/hr)	Carbon in Feed (wt%)	Mass Balance (%)	Carbon Balance (%)	Removal Efficiency (%)	Destruction Efficiency (%)
P560	16.5	1.75	75	2.2	6.6	95.5	134.0	19.3	85.7
P562	16.5	1.75	125	2.8	6.6	99.8	137.7	15.2	88.0
P564	45.5	3.25	75	1.8	6.6	100.2	143.0	36.0	92.7
P565	31	2.5	100	2.1	5.6	98.7	144.8	48.8	93.0
P566	16.5	1.75	75	2.4	6.4	100.0	130.2	4.9	96.0
P567	16.5	3.25	75	2.1	6.6	101.6	130.7	22.5	96.9
P568	45.5	1.75	75	1.6	4.3	100.8	126.2	64.1	82.9
P569	16.5	3.25	125	2.3	6.9	98.4	127.9	21.7	83.4
P572	31	2.5	100	2.3	5.4	98.8	147.9	37.9	81.7
P575	16.5	1.75	125	1.7	4.3	101.6	133.2	27.7	94.2
P576	16.5	3.25	125	2.3	4.2	100.0	136.6	29.0	97.0
P577	31	2.5	100	1.8	5.5	102.4	104.8	35.4	96.5
P580	45.5	3.25	75	2.6	4.3	102.8	120.2	57.3	89.0
P583	31	2.5	100	3.0	5.4	102.4	125.0	54.4	76.4
P584	31	2.5	100	2.2	5.4	100.6	135.5	39.9	89.2
P585	2	2.5	100	0.6	5.5	99.9	118.5	4.1	†
P588	31	1	100	2.4	5.4	100.0	104.7	55.6	92.1
P589	31	2.5	50	2.0	5.5	101.4	117.2	23.0	97.3
P590	31	2.5	100	2.0	5.5	100.9	92.0	45.4	97.1
P591	31	2.5	100	2.1	7.8	99.5	107.5	33.1	95.2
P593	31	2.5	150	2.2	5.4	102.4	128.5	47.2	82.4
P594	31	2.5	100	2.3	5.4	99.7	116.8	39.2	88.8
P595	31	4	100	2.3	5.4	101.1	144.1	39.9	81.4
P596	31	2.5	100	1.5	5.4	103.4	128.3	40.5	82.1
P638	16.5	3.25	75	1.6	4.4	100.5	149.5	38.2	87.2
P639	45.5	1.75	125	1.5	6.5	95.2	132.0	62.1	80.8
P640	45.5	3.25	125	1.6	4.3	101.3	112.1	56.7	90.9
P641	45.5	1.75	75	2.0	6.6	99.7	114.1	54.3	78.8
P642	45.5	1.75	125	2.2	4.2	100.4	108.1	76.2	83.5
P644	31	2.5	100	1.8	3.0	102.7	129.9	47.7	95.9
P645	60	2.5	100	2.1	5.4	98.9	127.9	67.3	85.7
P647	45.5	3.25	125	2.2	6.6	103.9	136.7	57.0	84.9

* All tests were performed using decolorizing carbon as the contaminant and SiO₂ as the inert matrix.

† Short residence time resulted in too small a gas sample to permit this calculation.

differences less than 1 mol% for all components. Instrument error for the GC is ± 2 mol%. The GC detection of water in the calibration gas is an analytical anomaly, the cause of which has yet to be defined but should diminish as the project progresses to larger-scale equipment and samples.

The results summarized in Table 1 show that it is possible to accurately track the fate of the carbon species in the LTP system. Carbon balances were calculated for each of the tests and range from 92.0% to 149.5%. The mean carbon balance of 126.4% is close to the median value of 128.3%, indicating that the values are tightly grouped and therefore representative. Although these balances appear to be somewhat high when considering that a perfect balance would be 100%, all are within analytical instrumentation error of 100%.

The product gas composition was calculated for each test and is summarized in Table 2. By far the most prevalent species found was oxygen. This is logical since excess oxygen was provided for reaction. Carbon dioxide (the primary product of the reaction of oxygen and carbon), carbon monoxide (the expected PIC), water vapor, and hydrogen were also found to be present. The GC was calibrated to detect hydrogen sulfide and various hydrocarbon gases, but as none were detected these results were not included in the table. A minute amount of methane was detected in the product of the test having the largest contaminant loading in the feed sample.

The reproducibility of LTP processing can be seen by comparing the product gas composition of Tests P572, P584, and P594. These tests were performed at nominally the same operating conditions and contaminant loadings. As Table 2 shows, the product gas compositions of these tests are nearly identical, indicating that the results are reproducible.

Contaminant removal and destruction efficiencies were calculated for each of the tests, even though it was not the goal of the Phase 1 testing to maximize these efficiencies. These values are presented in Table 1. Removal efficiencies ranged from about 4% to about 76%, while destruction efficiencies for the removed contaminants ranged from about 76% to about 97%. These encouraging results imply that, when operating conditions are optimized, it should be possible to meet the project goals of removal of at least 99 wt% of the contaminants and destruction of 99.99 wt% of the removed contaminants.

The statistical analysis has not yet been completed. It is expected that the statistical results will indicate which operating conditions should produce the highest contaminant removal and destruction efficiencies. Such information will be used in upcoming project phases.

CONCLUSIONS

Several conclusions can be drawn from the results:

- It is possible to obtain mass balance closures of ± 5 wt% with the existing LTP system.
- The analytical scheme currently in place accurately analyzes both solid and gaseous products.
- The fate of the contaminant species can be tracked in detail.
- The gaseous products of all tests consisted primarily of excess oxygen, carbon dioxide, and carbon monoxide.
- The tests are very reproducible.

In general, it can be said that both the mechanical and analytical aspects of the LTP system met or exceeded expected performance standards based upon previous research at the EERC.

TABLE 2. PRODUCT GAS SLATES FOR PHASE 1 TESTS

Test No.	O ₂ (mol%)	CO ₂ (mol%)	H ₂ (mol%)	H ₂ O (mol%)	CO (mol%)	CH ₄ (mol%)
P560	71.2	19.1	1.0	2.4	6.3	0.00
P562	77.2	16.4	0.8	2.1	3.5	0.00
P564	89.9	7.6	0.8	1.3	0.4	0.00
P565	85.2	11.2	0.7	1.7	1.3	0.00
P566	87.9	9.5	0.3	1.7	0.6	0.00
P567	91.5	6.3	0.5	1.4	0.3	0.00
P568	88.1	8.2	0.6	2.4	0.8	0.00
P569	70.3	20.4	1.4	1.6	6.3	0.00
P572	75.8	15.3	0.9	2.6	5.4	0.00
P575	81.4	14.7	0.7	1.9	1.4	0.00
P576	88.2	9.4	0.5	1.4	0.5	0.00
P577	91.5	7.5	0.6	0.0	0.4	0.00
P580	86.3	9.7	0.7	1.1	2.2	0.00
P583	80.0	13.5	0.5	0.9	5.1	0.00
P584	79.3	16.3	0.7	0.6	3.1	0.00
P585	99.6	0.0	0.0	0.4	0.0	0.00
P588	84.3	12.6	0.7	0.8	1.7	0.00
P589	92.9	5.6	0.4	0.9	0.2	0.00
P590	85.3	12.8	0.4	0.8	0.6	0.00
P591	80.0	16.2	0.4	2.1	1.3	0.02
P593	70.3	20.9	0.9	0.9	7.0	0.00
P594	78.5	16.4	0.5	1.4	3.2	0.00
P595	71.9	19.6	0.5	1.0	7.1	0.00
P596	68.5	22.0	1.2	0.8	7.5	0.00
P638	83.2	10.9	0.7	2.7	2.4	0.00
P639	70.4	18.9	0.6	3.2	7.0	0.00
P640	79.7	13.2	0.5	4.7	2.0	0.00
P641	93.0	3.9	0.5	2.3	0.2	0.00
P642	74.6	14.9	0.6	2.9	7.0	0.00
P644	83.6	10.5	0.4	4.8	0.7	0.00
P645	83.6	10.5	0.4	4.8	0.7	0.00
P647	73.9	17.6	0.7	2.9	4.9	0.00

FOR MORE INFORMATION

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A SUMMARY OF TWO RECYCLING TECHNOLOGY EVALUATIONS

COMPLETED UNDER THE MITE PROGRAM

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INTRODUCTION

Through the Municipal Solid Waste Innovative Technology Evaluation (MITE) Program, EPA has evaluated a number of promising innovative technologies for municipal solid waste (MSW) recycling. These technologies have varied from collection and processing methods to evaluations of products made from post-consumer material. The MITE program was conceived with the purpose of providing objective, third-party evaluations on environmental performance and costs of innovative municipal solid waste management technologies and this program is administered for EPA by the Solid Waste Association of North America (SWANA), representing the public sector solid waste management needs. The technologies are selected via an annual solicitation and review by an Advisory Group made up of recycling coordinators and solid waste managers.

Once the technologies are selected and the evaluation is performed, the results are published in a report that is distributed to the information users or the general public. The benefit for technology developers in participating is that they receive objective, published information to assist them in the commercialization and marketing of their technology.

One unique point separating this innovative environmental technology evaluation program from others is that it is often the public sector (the municipality, county, or solid waste district) advancing MSW technology through research and development, and partnerships with private companies and trade organizations. A significant number of applicants to the MITE program have been cities or non-profit groups. This is the case with the two evaluations profiled below.

This summary will address two evaluations focusing on the collection of municipal solid waste and designs that foster cost-effective recycling and source reduction.

METHODOLOGY

The first project profiled is a weight-based municipal solid waste collection system. The second project is an evaluation of three co-collection systems for solid waste and recyclables.

Weight-Based Collection Pilot in Farmington, Minnesota

The City of Farmington, Minnesota applied to the MITE program for an evaluation of their proposed pilot for a weight-based MSW collection system. The purpose of this collection system is to charge waste generators according to the weight of the solid waste placed at the curb for disposal. The more waste generated, the higher the disposal costs would be. There is an economic incentive in generating less waste for disposal and Farmington theorized that this type of system would increase the amount of material set out for recycling, a service for which there is no charge, and also foster source